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AUTOMATIC AJUSTMENT METHOD TO FIT T•LOG(T/τ_0) SCALING FOR TIME EVOLUTION THERMAL DEPENDING PROPERTIES OF NANOCRISTALLINE IRON

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Summary. In many forming processes the evolution of the material properties evolves with time and changes as temperature varies, forcing the use of a time-dependent thermomechanical computational model to simulate those processes. If the property under study could be described as a collectivity of microentities jumping over a certain distribution of energy barriers from one metastable state to another, then the whole behaviour of the material property follows a T·Ln(t/τ_0) scaling. The use of T·Ln(t/τ_0) as an integration variable could be employed to simplify the computational procedure. Also this scaling description allows to extrapolate the time evolution behaviour at times (or speeds) that are experimentally inaccessible. From this scaling it is also possible to determine the attempt period τ_0, needed by the computational model. All the properties described by the Arrhenius Law could be scaled by this method.

In the present work the results of appling this scaling method to the hardness of a nanocrystalline iron powder with several annealing treatments are presented, and an algorithm for automatic adjustment of a T·Ln(t/τ_0) scaling is tested.

INTRODUCTION

There is a broad variety of physical systems that show a time-dependent behaviour of some of their physical properties because of the existence of energy barriers, which separate local minima corresponding to different equilibrium states of the system. In recent decades, considerable effort has been devoted to understanding the relaxation phenomena in systems with different degrees of disorder, such as: high temperature superconductors[1] in which vortices are submitted to pinning by defects and dislocations, and where the application of a magnetic field creates metastable states in the vortex lattice; small magnetic particle systems[2] with a volume distribution and randomly orientated easy axes, which show blocking phenomena depending on the experimental time windows, and hardness and grain size time evolution of heat treated single phase materials[3] were atoms and dislocations are submitted to pinning. The existence of a scaling that allows to plot all the relaxation data

obtained at different temperatures in a unique master curve would make possible to cover a large number of time decades, due to any change in the temperature of the system corresponds to a change in the time scale of the relaxation.

The simplest case for the scaling law of a relaxing system governed by thermally activated processes is the so called $T \cdot Ln(t/\tau_0)$ master curve[4] . Due to there is not a known function for fitting, all approximation to the master curve must be subjective and hence the optimal value changes when the operator is changed. In the present work, a not subjective scaling method is presented and its validity is shown scaling the hardness evolution of nanocrystalline iron powder with several annealing treatments.

RELAXATION CURVES

The variation of a property for the assembly of weak (or non) interacting entities with a distribution of energy barriers $f(E)$, which are overcome by thermal fluctuations, according to the Arrhenius Law, is given by[2] :

$$\Delta P(t) = \Delta P_0^{\infty} \cdot \int_0^{\infty} dE \cdot p(t,E) \cdot f(E) \tag{1}$$

where P is the property under study, ΔP_0^{∞} is the total variation of the property between the initial and the equilibrium state, E is the energy barrier height and the function $p(t,E)$ is given, according to Neel's theory[5] , by:

$$p(t,E) = e^{-\frac{t}{\tau_0} \cdot \exp\left(-\frac{E}{k_B \cdot T}\right)} \tag{2}$$

where τ_0 is the attempting characteristic time and k_B the Boltzmann constant.

Taking into account that $p(t,E)$ varies abruptly from 0 to 1 as the energy barrier E increases for a given time t, is usual to simplify it by a step function with the discontinuity in $E_c(t)$, the inflexion point of $p(t,E)$, i.e.:

$$E_c(t) = k_B \cdot T \cdot Ln(t/\tau_0) \tag{3}$$

Then, equation (1) could be rewritten as follows:

$$\Delta P(t) = \Delta P_0^{\infty} \cdot \int_{E_c(t)}^{\infty} dE \cdot f(E) \tag{4}$$

where the only parameter of the evolution is the scaling variable $T \cdot Ln(t/\tau_0)$.

The validity of the scaling is determined by the validity of the step approximation, which is acceptable as long as the width of $p(t,E)$ was small compared to the width of the energy barrier distribution, $f(E)$[4] . This assumption is usually certain when, under experimental conditions, the time evolution of the system is clear enough.

FITTING METHOD

Nevertheless $f(E)$ and its integral are unknown, both must be quite soft functions far from

first order transitions. In this case, the integral can be locally approximated by its Taylor series, and this series estimated by a local spline. Assuming those premises, an error function ($F(\tau_0)$) could be calculated by:

$$F(\tau_0) = \sum_i \left[H_i - S\left(T_i \cdot Log\left(\frac{t_i}{\tau_0} \right) \right) \right]^2 \quad (5)$$

where H_i, T_i and t_i are the experimental hardness, temperature and time (respectively) of the sample i, and $S(x_i)$ the local spline at the i point. As the order of $T \cdot Ln(t/\tau_0)$ points depends on the τ_0 value, these points must be sorted each time that $F(\tau_0)$ is computed.

Using a standard numerical minimization algorithm, a local minima of $F(\tau_0)$ could be found. Running the procedure from several random distributed starting points, the global minimum can be reached, and deuce the τ_0 optimal value.

EXPERIMENTAL RESULTS

The powder was hardened in a planetary ball mill using the technique known as Ball Milling (BM). Within each grinding bowl were introduced 6,00 g of iron powder plus 1.2 wt% of ethylene-bis-Stearamide (EBS), 40 chrome steel balls of 10 mm of diametre. The weight relationship between balls and iron powder was 27:1. An argon overpressure applied to the recipients was used to prevent the powder oxidation during the milling.

The mill rotational speed was set at 160 rpm, with a 30 minutes running cycle followed by 30 minutes of stop period to prevent excessive heating of the powder. After 52 hours of efective working time, a 5 μm particle size and 8 nm crystal size powder was obtained[6] . Five series of 10 samples were prepared with the iron powder. Each sample was prepared with ~0.1 g of iron powder sealed in a glass tube under He atmosphere to prevent oxidation. Those samples were thermal treated at different temperatures (between 450 ºC and 700ºC) during different time (ranging from 1h to 2 days). Eleven Vickers indentations HV0.01 (ISO 6507-1) were carried out in each sample and the average value was taken as hardness value.

After that, the fitting procedure were applied with a cubic spline with 13 points (from i-6 to i+6 point) and reporting an attempting period $\tau_0 = 1.76 \cdot 10^{-5}$ s . In figure 1 the hardness of samples are plotted vs $T \cdot Ln(t/\tau_0)$ for this value, showing a good arrangement of the points.

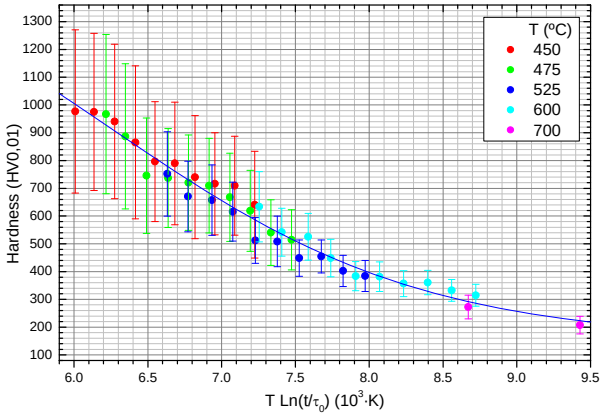

Figure 1: Scaling plots and fitting curves of nanocrystalline iron powder for $\tau_0 = 1.76 \cdot 10^{-5}$ s.

CONCLUSIONS

The $T \cdot Ln(t/\tau_0)$ scaling can reduce the cost of mechanical characterization of materials by the reduction of experimental points required to be measured.

The $T \cdot Ln(t/\tau_0)$ scaling could reduce the computational effort of simulations by the reduction of variables involved in calculus of mechanical properties.

The $T \cdot Ln(t/\tau_0)$ scaling can extrapolate results to time (or speed) experimentally unreachable, by a single change in the temperature of the experimental device.

The $T \cdot Ln(t/\tau_0)$ scaling can done by a nonsubjective algorithm.

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